SOP No. 29

Standard Operating Procedure
for the
Assignment of Uncertainty

1. Introduction

1.1. Purpose
Laboratories performing calibrations that meet ISO/IEC 17025 must report uncertainties in conformance with the *ISO Guide to the Expression of Uncertainty in Measurement (*hereafter called the *GUM)*. This SOP provides instructions for the laboratory to meet this requirement.

1.2. Prerequisites

1.2.1. Calibration certificates with valid uncertainties must be available for all standards.

1.2.2. Statistical data regarding the calibration measurement process must be available; preferably from measurement control programs within the laboratory.

1.2.3. Knowledge of the technical basis for the measurement is critical for completeness in uncertainty evaluation. This can be obtained through reference papers, reference procedures, brainstorming, experimentation, interlaboratory comparisons, cause and effect diagrams and the like. Each NIST SOP published in NISTIR 6969, 5672, and 7383 includes detailed uncertainty budget tables that may be used.

2. Methodology

2.1. Scope, Precision, Accuracy

Each measurement made in a laboratory has a corresponding uncertainty assigned to the calibration value. The uncertainty is directly related to the measurement parameter (scope), range of the measurement, the equipment or measurement process being used (affecting precision), and the standards available with associated uncertainties.

2.2. Summary

This uncertainty analysis process follows the following eight steps:
1) Specify the measurement process;
2) Identify and characterize uncertainty components;
3) Quantify uncertainty components in applicable measurement units;

4) Convert uncertainty components to standard uncertainties in units of the measurement result;
5) Calculate the combined uncertainty;
6) Expand the combined uncertainty using an appropriate coverage factor;
7) Evaluate the expanded uncertainty against appropriate tolerances, user requirements, and laboratory capabilities; and
8) Report correctly rounded uncertainties with associated measurement results.

Special methods for handling bias/errors and uncertainties associated with the use of multiple standards are addressed as well.

3. The Process of Measurement Uncertainty Estimation

3.1. Step 1. Specify the process.

Clearly specify the measurement process in question, including the item being measured and the input quantities upon which it depends. This will usually require a quantitative expression related to the process (i.e., a measurement equation). Where possible, you may reference an SOP or other method description along with the specific standards and measurement assurance process that is used to adequately complete this step.

3.2. Step 2. Identify and characterize uncertainty sources.

Identify all possible sources of uncertainty in a comprehensive list, characterizing them based on the evaluation method that will be used to quantify them (Type A, statistical methods or Type B, scientific judgment) and to categorize them based on their relatedness with something such as an uncertainty budget table.

Table 1. Example uncertainty budget table.

Uncertainty Component Description	Symbol	Source	Type (A or B)	Typical Distribution
Standard uncertainty from the measurement *process*	s_p	Process	A	Normal
Standard uncertainty for the *standards*	u_s	Cal Cert	B	See Cert
Standard uncertainty due to *other* factors	u_o	Analysis or References	B	Varies

Using the measurement equation that was identified in 3.1 provides a good starting point as do the detailed uncertainty budget tables provided in many procedures. All of the parameters in this expression may have an uncertainty associated with them. When there are discrete steps in the measurement process, additional uncertainties may be associated with each.

What follows are the most common uncertainties associated with metrological measurements. Keep in mind that this list is not exhaustive. Each item listed below is identified as a standard uncertainty, u, when determined using Type B methods of evaluation (technical judgment, theoretical assessments, reference items) and a standard uncertainty, s, when determined with Type A methods of evaluation (statistical methods). Each standard uncertainty is represented by a lower case variable and further defined by a subscript that is arbitrarily assigned and usually related to the source for ease in remembering that source.

3.2.1. Standard uncertainty from the measurement *process*, s_p, (Type A evaluation).

3.2.1.1. Standard deviation from a measurement assurance chart or control chart.

The value for s_p is obtained from the control chart data and the current knowledge that the measurements are in a state of statistical control. This must be ascertained by measuring at least one check standard during the course of the current measurements combined with data accumulated using the same process in previous measurements. The control chart data must reflect the measurement process being performed.

3.2.1.2. Standard deviation from a series of replicate measurements.

Measure a stable test object at least seven times, no two measurements of which should be made on the same day. Calculate the standard deviation in the conventional manner to obtain the standard deviation of the process, s_p, keeping in mind that it does not fully represent the measurement process under all typically encountered conditions and that additional uncertainty values may need to be addressed more fully.

Note: Repetitive measurements made on the same day estimate the short-term standard deviation of the process and may underestimate actual measurement variability.

3.2.1.3. Standard deviation when a process standard deviation is "zero".

When the standard deviation of a measurement process is less than the resolution of the measuring instrument, d, the standard deviation must be assessed. Generally, the smallest standard deviation to be used in this instance may be estimated as 0.577 times the instrument resolution from the equation noted below, with d being the instrument resolution. Assess the standard deviation and use the larger of the calculated standard deviation or the value from this equation. (Some procedures will provide for this value to be reduced further through repetitive measurements (e.g., SOP 4 and SOP 5); however, analysis of laboratory measurement assurance check standard data must be performed and be acceptable, to reduce the standard deviation of the measurement process further.)

$$s_p = \frac{d}{\sqrt{3}}$$

3.2.2. Standard uncertainty for the *standards*, u_s (Type B evaluation).

3.2.2.1. When using standards calibrated by another laboratory.

The information for the standards comes from the calibration report, generally reported as an expanded uncertainty with its coverage factor (k). The expanded uncertainty is simply divided by the stated k value to obtain the combined uncertainty for the standard, u_c, which represents the u_s when used in your laboratory. When k is not equal to two, additional factors associated with the degrees of freedom must be included. See Appendix A to determine the degrees of freedom associated with coverage factors. Keep track of the degrees of freedom for later use.

3.2.2.2. When using a standard calibrated in your laboratory (Type B evaluation).

If the standard was calibrated in your own laboratory, calculate the combined standard uncertainty, u_c, at $k = 1$ and use that as the standard uncertainty for the standard, u_s.

3.2.2.3. When using more than one standard (Type B evaluation).

When more than one standard is used in a calibration, the standard uncertainty for each, u_{s1}, u_{s2}, u_{s3}, etc., is included in the RSS equation if the standards have had independent calibrations. Standards with independent calibrations are combined by standard root sum square methods.

When calibrations are performed at the same time with the same reference standards, or when a standard is used multiple times, the standards are likely considered dependent, so the standard uncertainties are added ($u_{s1} + u_{s2}$) to determine a value to represent u_s. (This is the case with two 1 kg standards that were calibrated at the same time using a weighing design and subsequently used together as standards (restraints) in a weighing design. It is also the case when a 25 gal standard is used four times to calibrate a 100 gal standard.) There are also circumstances where both approaches are used to combine uncertainties from multiple combinations of standards.

3.2.3. Standard uncertainty due to *other* factors, u_o (Type B evaluation.)

These are factors related to the measurement equation, but distinct from the standard uncertainties associated with the process and the standards. These items are often much smaller in a well-controlled process than the standard uncertainties associated with the process and the standards. Examples are given in the uncertainty budget tables in each calibration SOP. Each component that is considered is included as an additional standard uncertainty u_{o1}, u_{o2}, u_{o3}, etc., and included in the RSS equation when data shows these factors to be significant. Documentation of the assessment of each component must be maintained to complete the documentation required by the specific calibration procedure and ISO/IEC 17025.

Additionally, the laboratory should include any other components that are considered significant. An estimated standard uncertainty that impacts the least significant digit in the combined uncertainty is considered significant. Additional uncertainties are addressed in the uncertainty budget tables provided in the SOPs.

3.2.4. Standard uncertainty due to factors *unrelated* to the measurement process per se, u_u.

These are factors that may be related to characteristics of the items being tested or of the standard and are usually minimized in well-known and controlled measurement processes. Review the uncertainty budget tables in each SOP or applicable international/national procedures or reference papers for more information.

3.2.5. Special uncertainties from other sources (Type B evaluations). Includes bias or unidentified errors.

It is a general requirement of the GUM that corrections be applied for all recognized and significant systematic effects and potential errors. Where a correction is applied based on a bias, an estimate of the associated uncertainty must then be included in the uncertainty analysis. Due to the various approaches present in the metrology system, several examples and possible approaches are presented in the section on calculating the combined or expanded uncertainties. At this stage, a determination must be made with regard to 1) identifying cause and 2) level of significance.

3.2.5.1. Identifying bias (offsets) and cause.

Bias or measurement offsets from reference values are often noted on control charts when an independent reference value is available and from results in proficiency testing. Control chart values are obtained over time and usually have more degrees of freedom than results from one or a few proficiency testing results. Therefore, noted offsets on control charts are more reliable estimates of bias when independent reference values are available. Evaluation of data from multiple sources should be considered whenever available (e.g., a recent calibration, a proficiency testing result, and control chart data assessed as a part of an integrated system).

If the cause of bias or error can be identified, it is usually corrected or applied to the measurement equation. In some cases, it is not possible to unarguably define the cause without exhaustive studies that provide little benefit. In those cases, the significance level *must* be evaluated before incorporating this type of an uncorrected systematic error in the calibration uncertainties.

3.2.5.2. Significance level.

When there is little to be gained from exhaustive studies on the measurement process to identify bias or potential errors, a test of significance may be conducted to determine alternative approaches for incorporating the bias into the uncertainty calculations.

In deciding whether a known bias can reasonably be included in the uncertainty, the following approach may be followed:

3.2.5.2.1. Estimate the combined uncertainty without considering the relevant bias.

3.2.5.2.2. Evaluate whether or not the bias is less than the combined uncertainty (i.e., $bias < \sqrt{u_{\mathrm{s}}^2 + \mathrm{s}_{\mathrm{p}}^2 + \mathrm{u}_{\mathrm{o}}^2}$).

3.2.5.2.3. If the bias is less than the specified limit, it may be included in the uncertainty using one of several approaches that must clearly be communicated in the report.

3.2.5.2.4. If the bias is larger than the specified limit, the error must be investigated further and corrected prior to providing calibration data.

If the deviations show that a standard is out of control, it should not be used for calibration until corrective action has been taken and the value for the standard is verified as being within criteria limits.

$$criteria\ limit : \left| \bar{x}_{lab} - \mathrm{x}_{\mathrm{ref}} \right| < u_c$$

If these differences are smaller than the criteria limits, investigation and corrective action may be unrealistic. If the deviations are less than the surveillance limits shown above, and corrective action is not taken, the deviations may be included in the uncertainty statement following one of several options given in the following section provided that the resulting uncertainty meets the needs of the customer or application. In all cases, the method used to incorporate bias must be clearly reported.

3.2.5.3. Option 1. Adding the bias to the expanded uncertainty (e.g., used in PMAP software). In this case, the bias is simply added to the expanded uncertainty and is reported as such.

$$U + \text{bias} = \left(u_c * k\right) + \text{bias}$$

3.2.5.4. Option 2. When uncertainties for the laboratory data and the reference data are considered equivalent (e.g., laboratory data is compared to data from another laboratory having equivalent precision) the equation below may be used.

In this case, a rectangular distribution is considered where the value might possibly be anywhere within the range shown for each laboratory data point. This method is referenced in section 4.6 of NIST Technical Note 1297. This approach may also be used in the case where a standard is predictably drifting with use over time. In this case, a mid-range value is chosen and u_d (uncertainty for *differences*) is calculated as follows:

$$u_d = \frac{\text{bias}}{2}\frac{1}{\sqrt{3}} \quad \text{or more simply: } 0.29\text{ d, where d is the bias}$$

3.2.5.5. Option 3. When uncertainties for the laboratory data are considered secondary to a reference value (e.g., the difference between the laboratory data and data from a higher level calibration with smaller uncertainties) the equation below may be used.

In this case, a reference value is given precedence over the laboratory data and a mid-range value is not chosen. The extreme value is more probable. In this case, the bias is treated as an uncorrected systematic error and the following equation may be used:

$$u_d = \frac{\text{bias}}{\sqrt{3}} \quad \text{or more simply : } 0.577\text{ d, where d is the bias}$$

3.3. Step 3. Quantify uncertainty estimates

All uncertainty estimates identified in the previous step must be quantified in units that represent the measured values. Type A methods of evaluation usually provide quantified estimates in the units of interest.

Type B methods of evaluation may be conducted with spreadsheets using the basic expression identified in the SOP or identified when the process was specified. Scenario testing can be done to determine the impact and quantify specific variables on the final measured quantity. The knowledge gained in this step often proves useful in identifying potential areas of improvement especially if contributing factors are graphed in a histogram or Pareto chart.

3.4. Step 4. Convert all factors to standard uncertainties

In cases where the uncertainty factors were determined statistically (Type A methods), the standard deviation is used to represent the standard uncertainty. In other cases, estimates must be made to ensure that the quantified uncertainties represent "one-standard-deviation" values or a $k = 1$ coverage level.

The appropriate distribution factor must be used when converting estimated uncertainty values to standard uncertainties. According to NIST Technical Note 1297, a rectangular distribution is generally used when detailed information about the distribution is unknown.

$$u_n = \frac{\text{value}}{\sqrt{3}}$$

3.5. Step 5. Calculate the combined uncertainty

The combined standard uncertainty, u_c, includes the standard uncertainty reported for the standards used, u_s, the standard uncertainty of the measurement process, s_p, the standard uncertainty from other sources, u_o, which includes all other factors the laboratory considers significant, the standard uncertainty due to factors related to the measured item but unrelated to the measurement process, $u_{u,}$ and finally, the standard uncertainty due to bias or differences, $u_{d,}$ when u_d is included. The standard uncertainties are usually combined using the root-sum-of-the-squares (RSS) method as follows:

$$u_c = \sqrt{s_p^2 + u_s^2 + u_o^2 + u_u^2 + u_d^{\ 2}}$$

Table 1. Symbol descriptions.

Symbol	Description
U	Expanded uncertainty (this is represented with an upper-case U)
u_c	combined standard uncertainty
s_p	standard uncertainty (standard deviation) of the "process"
u_s	standard uncertainty of the "standard"
u_o	standard uncertainty of "other factors"

Symbol	Description
u_u	standard uncertainty of factors "unrelated" to the measurement process
u_d	standard uncertainty of "differences" (may be treated in different ways)
k	coverage factor

3.6. Step 6. Calculate the expanded uncertainty

The combined standard uncertainty is then multiplied by a coverage factor, *k*, based on the degrees of freedom, to provide a level of confidence of approximately 95 % or 99 %, respectively (depending on what is required by the customer, but most often 95 %). The equation used to determine the expanded uncertainty is as follows:

$$U = u_c * k$$

where $k = 2$ a value from the table in Appendix A, taken from the column 95.45. When there are a small number of degrees of freedom, the coverage factor must be determined from a statistical table such as provided in the Guide to the Expression of Uncertainty in Measurement or NIST Technical Note 1297 (See Appendix A).

Situations where the degrees of freedom associated with the value for a standard in the traceability chain require special care to ensure that an appropriate coverage factor is used for calculating the uncertainty of subsequent reported values. When there are a small number of degrees of freedom used to determine the standard deviation of the process and/or the *k* value from the calibration report represents a small number of degrees of freedom, the *effective degrees of freedom* must be used to determine an appropriate coverage factor. Use the equation provided in NISTIR 6969, Section 8 or the Welch-Satterthwaite equation provided in NIST Technical Note 1297 or the Guide to the Expression of Uncertainty in Measurement.

3.7. Step 7. Evaluate the expanded uncertainty

The expanded uncertainty must be evaluated. Evaluation may be against established criteria such as tolerance limits, customer requirements, and/or calibration and measurement capabilities listed on the laboratory scope. For example, the specifications for mass standards clearly state that the expanded uncertainty must be less than 1/3 of the tolerance.

3.8. Step 8. Report the uncertainty

Once the uncertainty has been calculated, round the value according to GLP 9, report the value, and include a statement such as the following is reported:

The combined standard uncertainty includes the standard uncertainty reported for the standard, for the measurement process, and for any observed deviations from reference values (e.g., from NIST), which are less than surveillance limits. The combined standard uncertainty is multiplied by k, a coverage factor of (insert value used) to give the expanded uncertainty which defines an interval with an approximate 95 % level of confidence.

The value selected for the coverage factor must be appropriate for the degrees of freedom available and the desired level of confidence associated with the uncertainty as noted in Step 7.

Appendix A

Value of $tp(\nu)$ from the t-distribution for degrees of freedom ν that defines an interval $tp(\nu)$ to $+tp(\nu)$ that encompasses the fraction p of the distribution. Note: Table is taken from NIST Technical Note 1297. The column marked 95.45(a) is generally used to give the "approximate 95 % confidence interval."

Degrees of freedom ν	Fraction p in percent					
	68.27(a)	90	95	95.45(a)	99	99.73(a)
1	1.84	6.31	12.71	13.97	63.66	235.80
2	1.32	2.92	4.30	4.53	9.92	19.21
3	1.20	2.35	3.18	3.31	5.84	9.22
4	1.14	2.13	2.78	2.87	4.60	6.62
5	1.11	2.02	2.57	2.65	4.03	5.51
6	1.09	1.94	2.45	2.52	3.71	4.90
7	1.08	1.89	2.36	2.43	3.50	4.53
8	1.07	1.86	2.31	2.37	3.36	4.28
9	1.06	1.83	2.26	2.32	3.25	4.09
10	1.05	1.81	2.23	2.28	3.17	3.96
11	1.05	1.80	2.20	2.25	3.11	3.85
12	1.04	1.78	2.18	2.23	3.05	3.76
13	1.04	1.77	2.16	2.21	3.01	3.69
14	1.04	1.76	2.14	2.20	2.98	3.64
15	1.03	1.75	2.13	2.18	2.95	3.59
16	1.03	1.75	2.12	2.17	2.92	3.54
17	1.03	1.74	2.11	2.16	2.90	3.51
18	1.03	1.73	2.10	2.15	2.88	3.48
19	1.03	1.73	2.09	2.14	2.86	3.45
20	1.03	1.72	2.09	2.13	2.85	3.42
25	1.02	1.71	2.06	2.11	2.79	3.33
30	1.02	1.70	2.04	2.09	2.75	3.27
35	1.01	1.70	2.03	2.07	2.72	3.23
40	1.01	1.68	2.02	2.06	2.70	3.20
45	1.01	1.68	2.01	2.06	2.69	3.18
50	1.01	1.68	2.01	2.05	2.68	3.16
100	1.005	1.660	1.984	2.025	2.626	3.077
∞	1.000	1.645	1.960	2.000	2.576	3.000

(a)For a quantity z described by a normal distribution with expectation μz and standard deviation σ, the interval $\mu z \pm k\,\sigma$ encompasses p = 68.27, 95.45, and 99.73 percent of the distribution for k = 1, 2, and 3, respectively.